

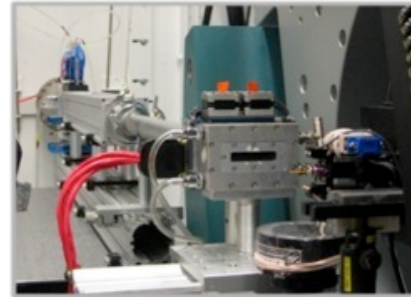
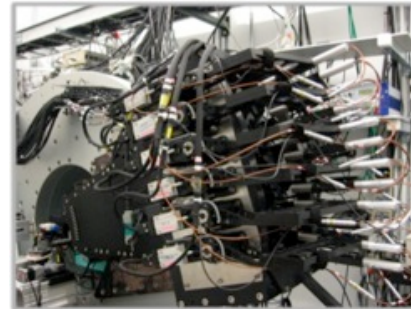
# Synchrotron Powder Diffraction Simplified: *Introducing the Advanced Photon Source's dedicated high-resolution beamline 11-BM*

**Matthew Suchomel**, Brian Toby, Lynn Ribaud, and Robert Von Dreele

*Advanced Photon Source, Argonne National Laboratory*

# Presentation Outline

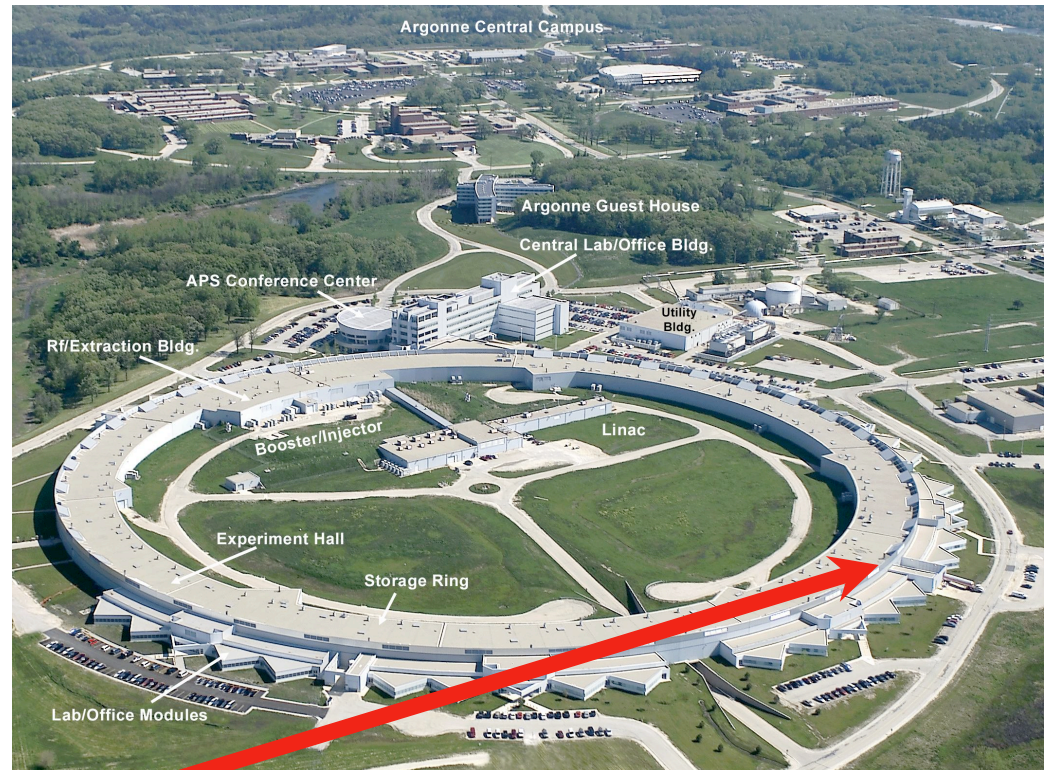
- Background to the APS & 11-BM
- High-Resolution Powder Diffraction
  - Why is it useful?
  - What do we offer?
- 11-BM Beamline “Tour”
  - with live-action robot movie!
- Performance & Examples
- Rapid Access Mail-in Program:
  - How does it work?
  - How can you get access?



**11-BM = Synchrotron Powder Diffraction Simplified !!**

# Argonne's Advanced Photon Source (APS)

- APS opened 1996
- On ANL campus
  - 30 min from Chicago
- 7 GeV ring current
  - ring circumference > 1 km
- 33 beamline ports
  - ~ 60 active stations

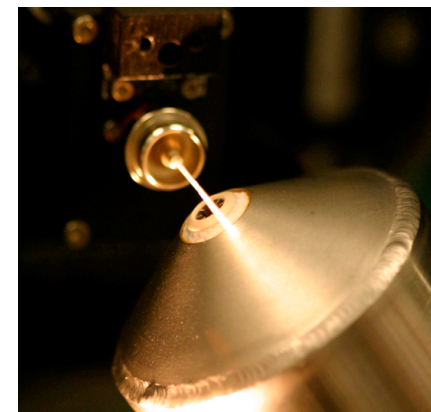
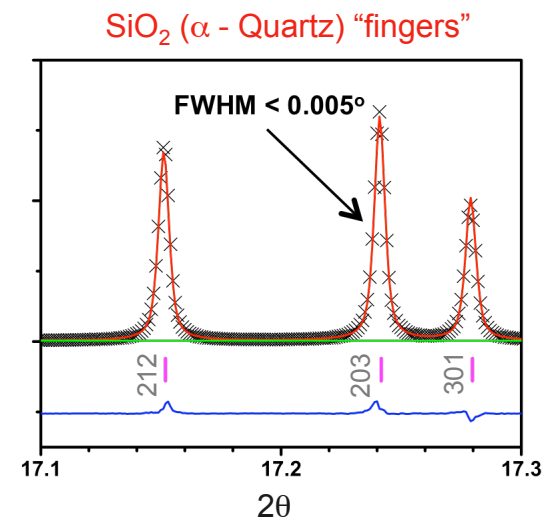


**High-Resolution  
beamline 11-BM**

- ✓ *started construction: spring 2005*
- ✓ *first beam in hutch: fall 2007*
- ✓ *mail-in user program: summer 2008*
- ✓ *first user publication: fall 2008*
- ✓ *on-site experiments: spring 2010*

# Instrument Overview

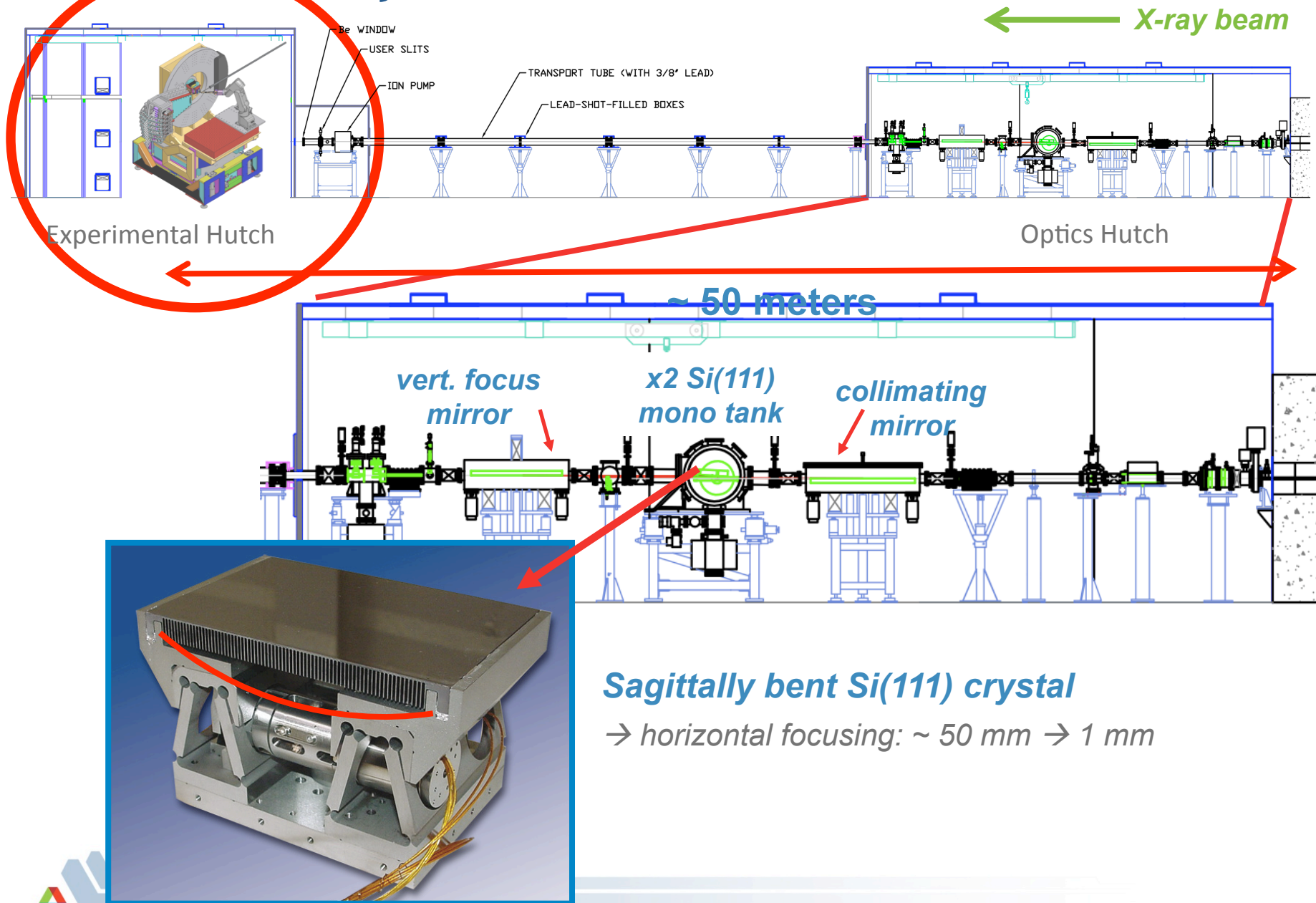
- Energy range (BM): 10 - 35 keV (1.0 - 0.35 Å)
  - mail-in service optimized @ 30 keV (~ 0.4 Å)
- Resolution:  $\Delta d/d$  ( $\Delta Q/Q$ )  $\approx 2 \times 10^{-4}$ 
  - Equivalent to best-in-world (ESRF, Diamond, PSI etc.)
  - Highest resolution powder diffraction in Americas
- Q range:  $Q_{\max} \approx 25 \text{ \AA}^{-1}$ 
  - 1 hr mail-in scan: 2Q range  $0.5^\circ \rightarrow 50^\circ$  ( $d \geq 0.5 \text{ \AA}$ )
- Beam Size: 0.5 mm (vert.) x 1 mm (horz.)
  - sample capillary < 1 mm  $\rightarrow$  min. axial divergence
- Multiple Sample Environments
  - Cryostream (90 K - 450 K), Hot Gas Blower (< 1000 °C)
  - *acquiring: 5 K Helium Cryostat, In-situ Pressure & Gas Cells*
- Robotic Arm  $\rightarrow$  Automated sample changes
  - Robot + Database  $\rightarrow$  High Throughput!



Hot Gas Blower in Action



# Beamline Layout



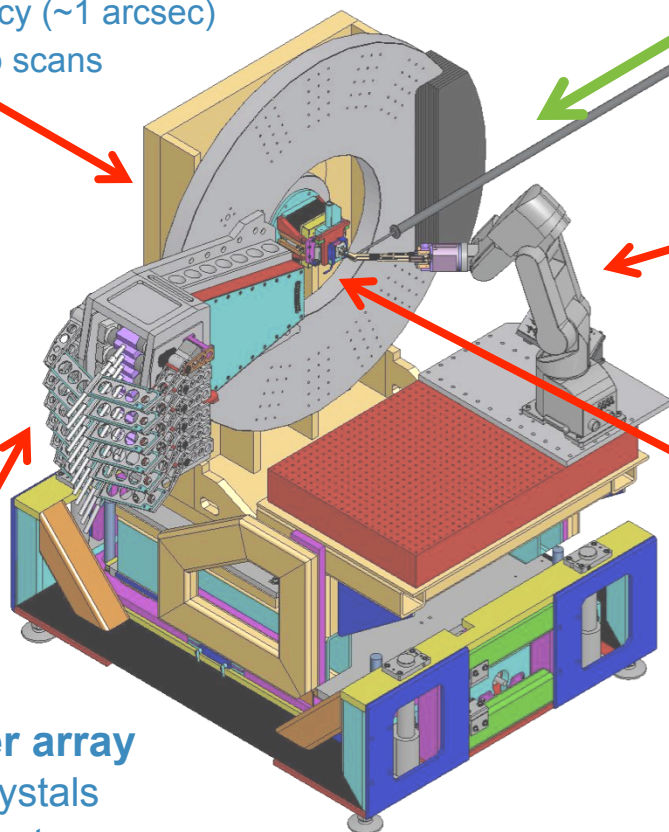
# 11-BM Diffractometer

## Huber 480 rotation stage:

high precision ( $\sim 0.35$  arcsec)

high accuracy ( $\sim 1$  arcsec)

slew or step scans

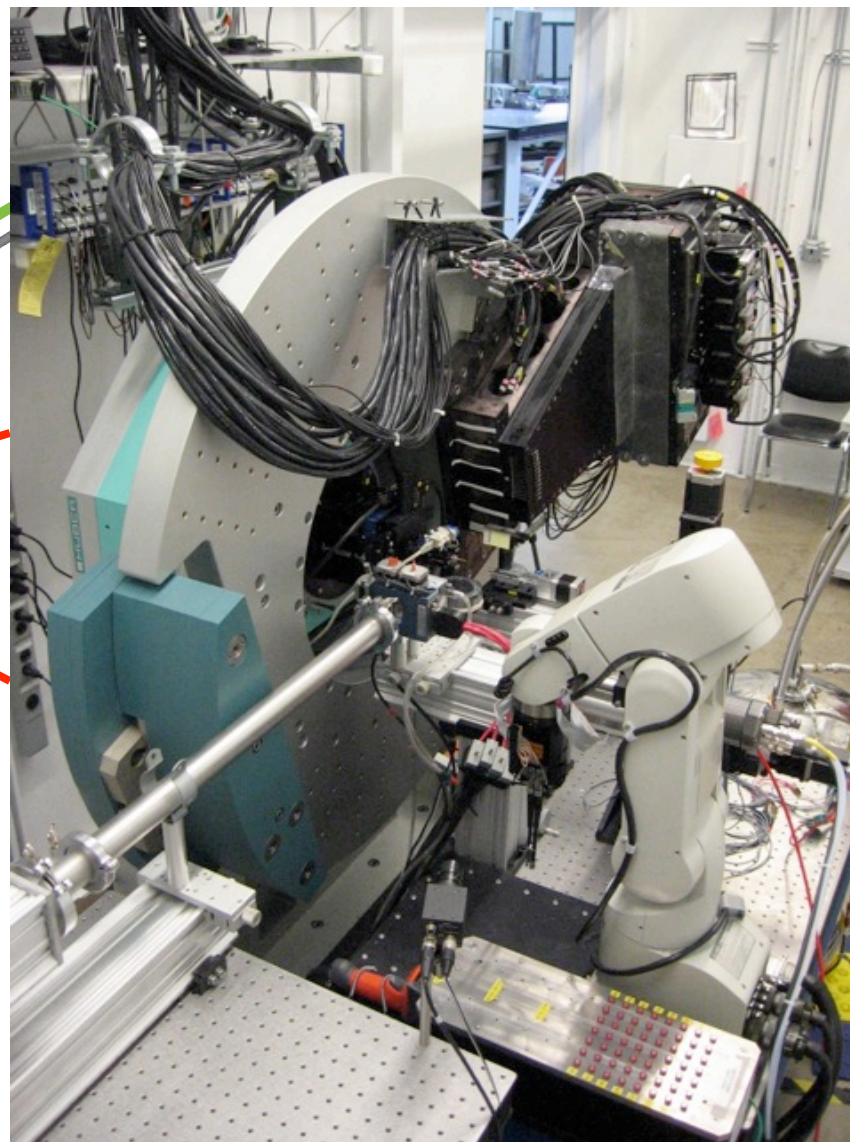


## 12 analyzer array

Si(111) crystals

LaCl<sub>3</sub> detectors

2° apart in  $2\theta$



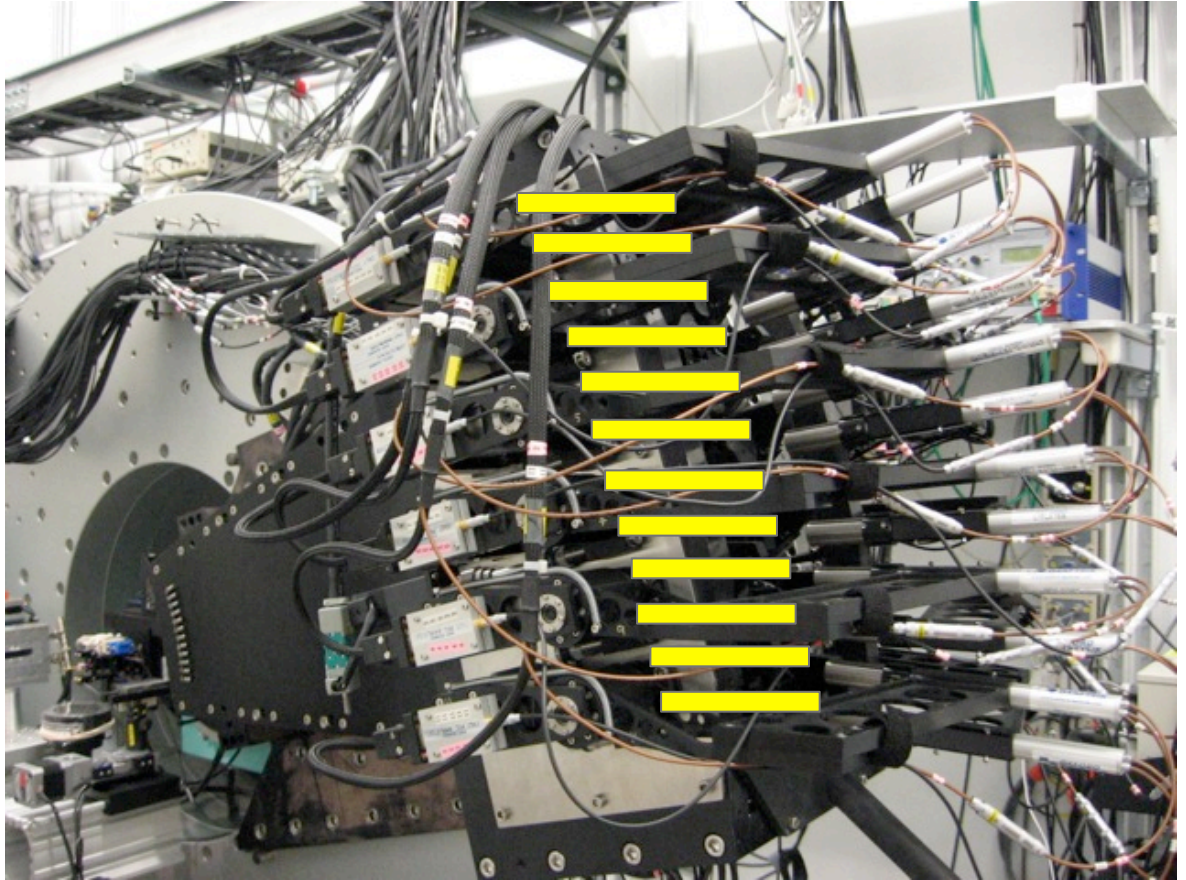


# 11-BM Detector Assembly

- 12 crystals/detectors
  - Individually adjusted
  - $2^\circ$  separation
  - Total  $2\theta$  range =  $22^\circ$
- Si(111) crystals
  - excellent discrimination
  - low background (& yield!)
  - $\text{LaCl}_3$  Scintillators

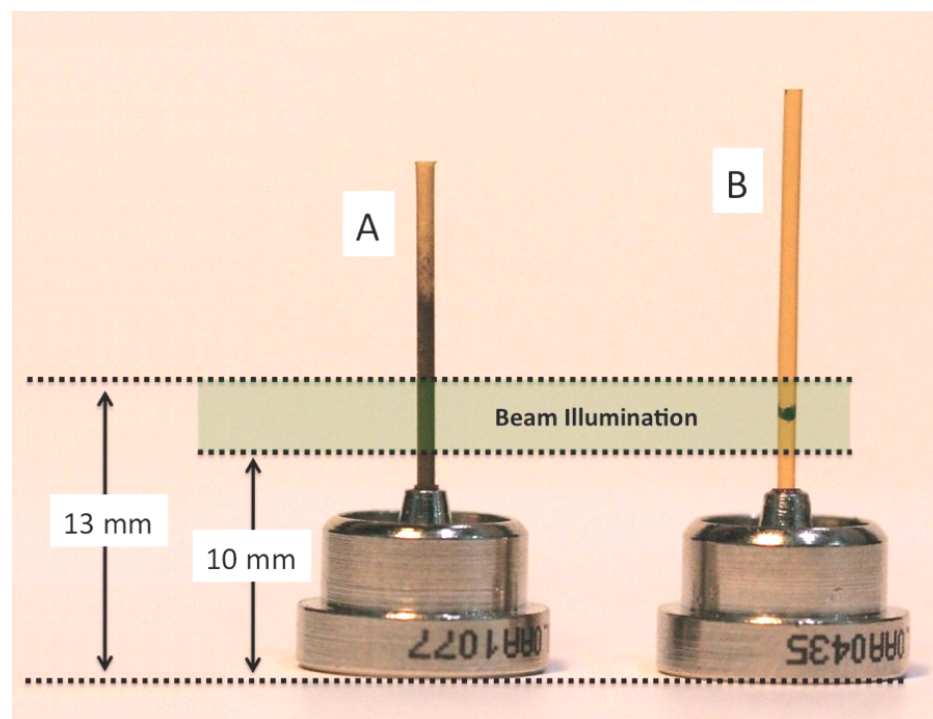
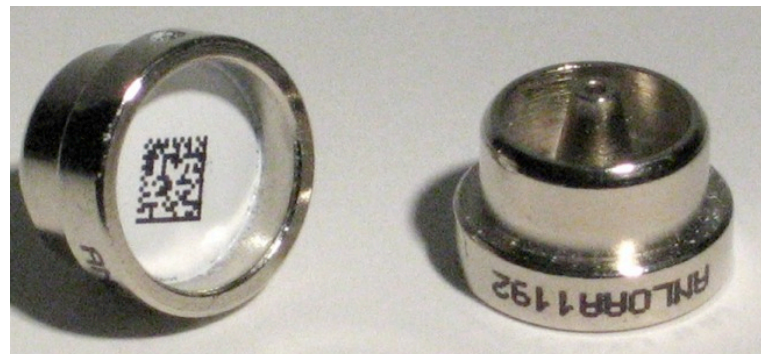
## Multiple Observations

- Reduce Collection Time
- Improve Accuracy



# Enabling High-Throughput

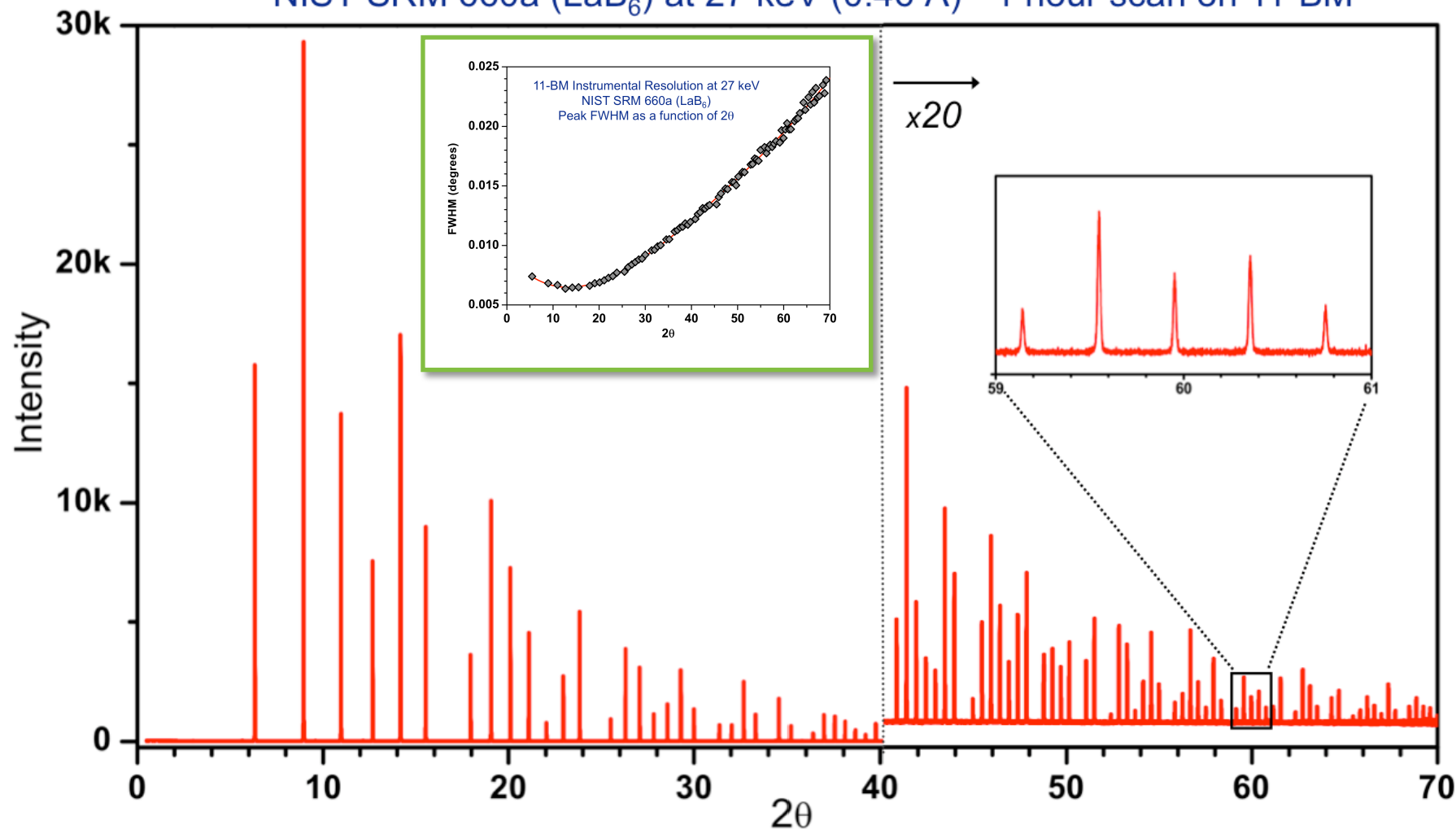
- Robotic Sample Changer
  - 100+ Samples
  - Works Nights & Weekends
  - Optical Barcode Reader
- Mail-in Sample Bases
  - Magnetic Base
  - Kapton® Capillary Tube (0.8 mm)
  - Unique Barcode for each Sample
- Software & Database
  - Automates Repetitive Tasks
  - Python, PHP/MySQL
  - Track Sample History & Detail
    - request -> receipt -> scan -> disposal*





# Performance

NIST SRM 660a (LaB<sub>6</sub>) at 27 keV (0.46 Å) - 1 hour scan on 11-BM



# How can YOU Access 11-BM ?

- Standard User Proposal
  - Multi-day experiment
  - On-site (or mail-in)
  - Additional sample environments (temp, atmosphere, pressure etc)
  - 3 deadlines/year, committee reviewed
- Rapid Access Mail-in Proposal
  - Same-day approval
  - 1 shift (~ 8 scans) limit
  - Selection of defined scans/temps
  - Notification & data receipt by email
  - Quick turn-around: Proposal -> Data in ~3 weeks
  - *Complements NPD studies (joint X-ray/Neutron refinements)*

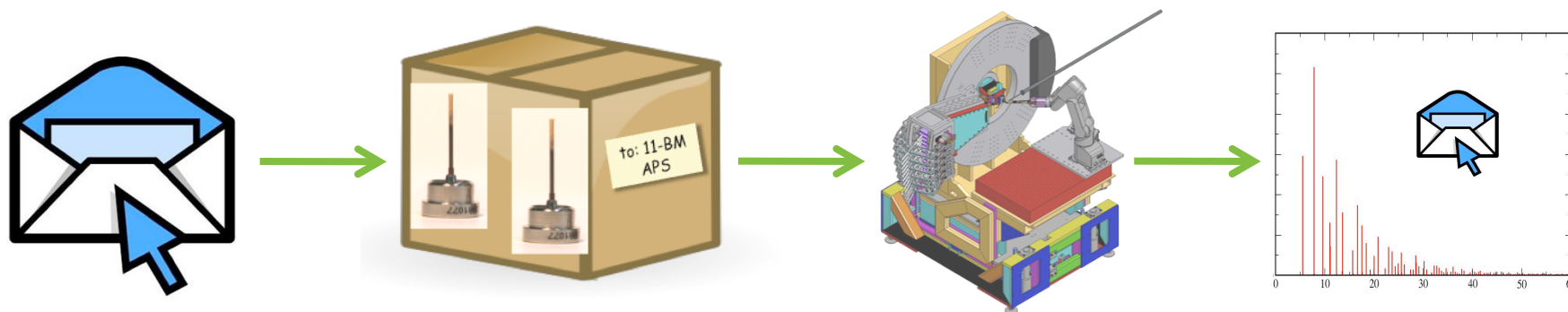


**6 easy steps for 11-BM rapid access**

# Mail-In Service Overview:

1. Submit rapid-access APS proposal for 11-BM beamtime
2. Proposal accepted → 11-BM staff send sample bases
3. User registers samples online & requests scan type/temp
4. User mails prepared samples back to APS
5. Data collected → email notification, user downloads data
6. Periodic reminders for analysis status & publication updates

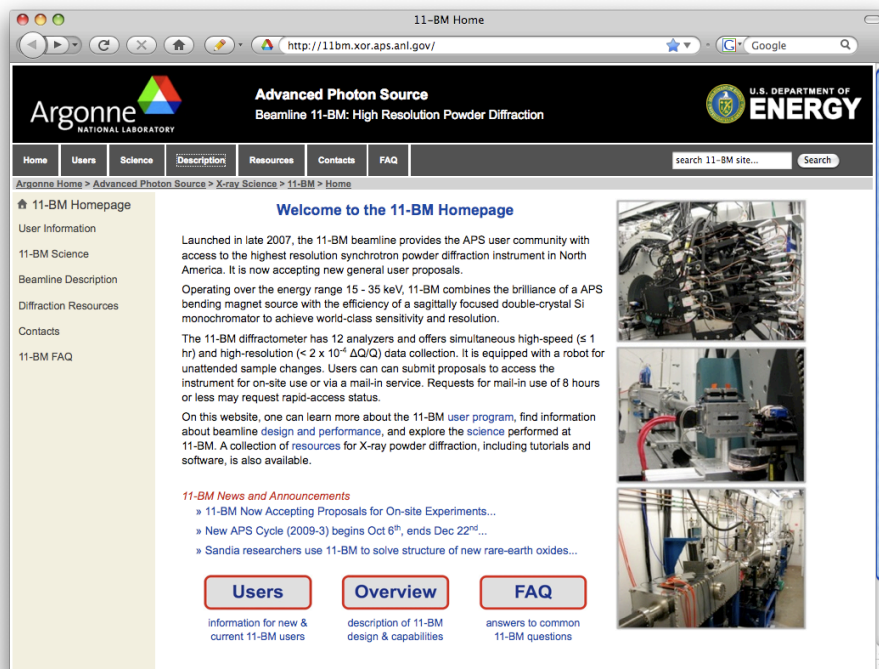
free service for all non-proprietary users



# More Information

11-BM webpage: <http://11bm.xor.aps.anl.gov/>

email contact: [11BM@aps.anl.gov](mailto:11BM@aps.anl.gov)



## 11-BM Acknowledgements

**11-BM staff:** Brian Toby, Lynn Ribaud, Bob Von Dreele and former staff: Jennifer Doebbler, Jun Wang & Sytle Antao.

**APS Support:** Peter Lee, Mohan Ramanathan, Chuck Kurtz, Curt Preissner, Xuesong Jiao and many many others...

**DOE grant proposal (2003)** by J.F. Mitchell, J.D. Jorgensen, R.B. Von Dreele, P.L. Lee, & M.A. Beno